



Fw: Comments--USGS Draft OFR
Elisabeth Evans to: Bonita Lavelle

01/12/2010 03:00 PM


1135497 - R8 SDMS

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----- Forwarded by Elisabeth Evans/R8/USEPA/US on 01/12/2010 03:04 PM -----

From: Greg Meeker <gmeeker@usgs.gov>
To: Elisabeth Evans/R8/USEPA/US@EPA
Date: 01/12/2010 11:50 AM
Subject: Re: Comments--USGS Draft OFR

Liz

The changes based on reviewers comments have been made and the report will go back to the authors today for final approval. After that, probably tomorrow or Thursday, the report goes to the publications group where it will get Bureau approval and be copy edited for posting. This could take as little as a couple of weeks or longer if someone finds an issue they feel is not properly addressed. As this is a relatively simple "data dump" without much interpretation, I don't think there will be any issues and the process should proceed fairly quickly. I will advise everyone that EPA is eager to have this published as soon as possible.

I should also let you know that there is much interest in this report from Mickey Gunter at U. of Idaho. Mickey was the mineralogy expert for W.R. Grace during the trial and I believe he is still working for Grace and maybe other organizations (NSSGA?) with an interest in contamination in Libby. Mickey has also done his own study (see attached).

Hope this helps,

Greg



Gunter_p837-840_09.pdf

On Jan 12, 2010, at 10:57 AM, Evans.Elisabeth@epamail.epa.gov wrote:

> Hi Greg
>
> Thanks for sending us information about the process to finalize the
> Draft OFR. We look forward to its completion.
>
> Please send us your best estimate for finalization and release of the
> report, including time budgeted for the formal scientific review
> process.
>
> liz
>
> Elisabeth Evans

-----Forwarded by Mary Goldade/R8/USEPA/US on 01/08/2010 11:11AM

To: <dadams@usgs.gov>
From: Mary Goldade <mary4pilates@hotmail.com>
Date: 01/08/2010 10:52AM
Cc: Mary Goldade/R8/USEPA/US@EPA
Subject: FW: Comments--USGS Draft OFR

Hey David,
I picked up your voice mail late yesterday pm. Arrgh! I'm
irritated that you didn't get this. I see I screwed up your

address. That's what happens when I work so late into the night. Ironically, I was trying to finish up before I left on vacation....
Please call me at home today to confirm your receipt of this.
970.224.2538

Can you call me to let me know when you think you can make changes? Someone mentioned off-handedly to me yesterday that

are having a mtg on Libby next week. I think they'd like to talk about this then, too. Please let me know what is feasible.
Thanks,
Mary

From: mary4pilates@hotmail.com
To: daddams@usgs.gov
CC: goldade.mary@pea.gov
Subject: Comments--USGS Draft OFR
Date: Thu, 17 Dec 2009 23:36:39 -0700

Hi David,
Whew, it's been a busy week, and my travel to Missoula this week was full of unexpected "travel challenges". ...Today was the crowning touch...I was dealing with a cancel flight that I had reschedule, etc...So, I'm a little frazzled...and rushed!

Regardless, as promised, I've performed a technical review on

Draft Open File Report. In general, it looks like the data are there, though I wanted to see more summary/background on the study purpose and it's description. However, I recall you

us in Nov. that you've structured the OFR to be simply a data output report. And, so, that may not be the intent of the

My comments are provided below. Note that symbols like um and uL will have to be replaced with greek "mu" symbol. Don't forget to include registration mark when you name a manufacturer (JOEL, Millipore, etc.). If you'd like me to provide recommended text for those into those sections I suggest need filling out, just let me know. I just don't want to craft language for you, if you're not interested in including it in the OFR. Thanks for your work on this. And, thanks you for the opportunity to review this document.

If you would, please advise when you've posted the final report. I know this is USGS' report and EPA doesn't have any say as to when/how things are completed , but there is great interest in

> the report at my office. I look forward to seeing the published.
> paper later in the Spring!

> I'm off for the next couple weeks. Have a great holiday!
> Mary Goldade
> Senior Environmental Scientist/Chemist

> 1. Introduction: (Note: some of these
> recommendations may not be appropriated for the purpose of
> your OFR)

> a. 1 st paragraph. Please cite the Study
> Design Document I prepared for planning and
> implementation of your work (USEPA 2008).

> b. Delete 2 nd sentence; it's not fully
> accurate

> c. 1 st paragraph, 3 rd & 4 th sentences.

> Move

> to Sampling section.

> d. 1 st paragraph. Consider revising this to
> something such as: "The purpose of this Open-File
> Report is to describe the findings of samples
> collected from three localities in Libby, Montana in
> June 2008. Vermiculite Mountain, located
> approximately 6 miles northeast of the town of

> Libby,

> contains winchite-richterite-tremolite amphibole
> (Meeker 2003). The U.S. Environmental Protection
> Agency (EPA) Region 8 is conducting remedial
> activities to characterize and remediate the
> amphibole (termed for convenience as Libby-type
> amphibole) present in and around Libby. As part of
> that investigation, EPA must determine background
> levels of Libby-type amphibole (LA). EPA requested
> the U.S. Geological Survey to collect and analyze
> samples in the region to determine if background
> levels of LA in the region are present which could

> be

> attributed to prehistoric glacial processes."

> e. 2 nd paragraph.

> i. Consider creating a separated
> section entitled "Sampling" and putting this
> paragraph there.

> ii. I recommend providing additional
> info about the field sampling like you do for
> the pits. For example I know that we removed
> and discarded the top organic layer of the
> field sample.

> iii. Please reference the sample
> locations in your description. For example,
> revise the 1 st sentence to read: "...sediment
> layers of two open pits (i.e., Sand Pit and
> Clay Pits) where sand..." and the 2 nd sentence:
> "Sampling was also conducted in an undisturbed
> field (i.e., Field) lying adjacent to the
> Wooden Field that was previously a source for
> fill material."

> iv. 4 th sentence. Replace "cross
> contamination" with "cross-contamination".

> 2. Sample Preparation:

> a. 1 st paragraph, 2 nd sentence. Replace
> "cross contamination" with "cross-contamination".

> b. 1 st paragraph, 2 nd sentence. Revise
> sentence to read: "...individually in a fume hood
> fitted with a HEPA (High-Efficiency Particulate Air)
> filter for a minimum..."

> c. 1 st paragraph, 5 th sentence. I recommend
 > removing the filler -word "then" from most of your
 > descriptions here.

> d. 2 nd paragraph, 1 st sentence. Consider
 > revising this paragraph to read: "Prior to milling
 > any samples and between each sample, the ball mill
 > was cleaned using several steps to avoid
 > cross-contamination. The ball mill was first
 > cleaned by..."

> e. 2 nd paragraph. Last sentence may be
 > deleted, if above recommendation is applied.

> f. 3 rd paragraph.

> i. Consider adding prior to 1 st
 > sentence: "The milled samples were mounted for
 > scanning electron microscope (SEM) analysis. "

> ii. 1 st sentence. Replace: "...in a
 > heap
 > filtered fume hood." with: "... in a fume hood
 > fitted with a HEPA (High-Efficiency
 > Particulate
 > Air) filter."
 > iii. Continuing. Revise to read:
 > "The
 > weighed sample was diluted with 125 mL water
 > (note: describe the nature of water—certified
 > fiber-free? Deionized?...laboratory water)
 > using an 800 mL graduated cylinder which was
 > continuously mixed using a magnetic stir bar.
 > A 45 uL aliquot of the suspension material
 > was drawn from the graduated cylinder using a
 > 15 uL pipette fitted with a 200uL pipette tip.
 > The material suspension was seated onto a 25
 > mm
 > diameter, 0.2 um pore size (polycarbonate?)
 > membrane filter using a clean Millipore
 > (insert
 > registration mark) filtration apparatus and
 > hand pump to ensure even distribution onto the
 > filter. The filter was attached to an aluminum
 > SEM stub using high-purity carbon tabs (is
 > there a manufacturer name?). Two SEM..."

> 3. Methods:

> a. Consider renaming this section "Analytical
 > Method"

> b. I would have expected that each of your
 > samples may vary in the number of FOVs
 > analyzed...depending upon the number of fibers found
 > in
 > each mag/FOV. However, this section appears to
 > indicate that you observed a standardized number of
 > FOV per mag. If this is not the case, please revise.
 > If it is the case, revise text as indicated below.

> c. 1 st sentence. Revise sentence to read:
 > "Each SEM stub was examined using 3 magnifications
 > at
 > 50X, 500X and 2000X in randomly selected,
 > non-overlapping fields of view (FOVs). A total of
 > 112
 > FOVs were examined: 12 FOVs at 50X, 50 FOVs at
 > 500X
 > and 50 FOVs at 2000X. Every FOV was inspected for
 > elongated(?) particles. If elongated(?),
 > cylindrical,
 > or suspicious particles..."

> d. Include the particle counting rules (eg 3:1
 > aspect ratio, minimum length/widths, etc)

> e. Include your analytical sensitivity and a
> description of uncertainty around the measured
> values.
> f. Reference the SOP used for preparation
> and
> analysis.

4. Results:

> a. 1 st paragraph, 1 st sentence: Replace
> "GPS" with "Global Positioning System (GPS)".
> b. This section appear to suggest a difference
> between LA and "non-fibrous amphiboles such as
> tremolite, actinolite..." Please clarify the
> distinction. I presume it is a difference in 3:1
> versus less than 3:1 ARs?
> c. 2 nd paragraph.
> i. 2 nd sentence. Report the LA levels
> here.
> ii. 3 rd sentence.
> iii. Consider including the elevations
> of LA detections in the Sand and Clay Pits
> iv. 4 th sentence. Revise sentence to
> read: "The final sample containing LA is from
> the Field was 20-22 inches below ground
> surface
> and at a concentration ranging from
> approximately 0.003 (or is it 0.004?) % to
> 0.05
> (or is it 0.047 ?)% by weight."
> d. Be consistent about significant figures,
> specifically about whether to report to 2 or 3 sig.
> figs.

5. Table 1.

> a. Consider including the Table number and
> title at the top of the table since it carries over
> to several pages.
> b. It may be my copy, but the table headers
> need to be repeated on subsequent pages.
> c. Revise the header term : "Notes" to
> "Comments" since you have table Notes.
> d. LA concs. Report ND or < (analytical
> sensitivity), but not 0.000%. Adjust reported values
> for sig. fig. comment (above)
> e. Notes. Please I didn't review this section
> carefully, but did note that the checking
> calculations did not include meters converted from
> feet like the other notes.

6. Conclusion.

> a. 1 st sentence. Revise the sentence to
> read:
> "Seventy samples representing discrete individual
> stratigraphic layers were collected from three
> localities in or near the town of Libby, Montana."
> b. 2 nd sentence. Consider revising the
> sentence to read: "...detectable LA. Measured
> concentrations were: [insert range and
> elevation]at
> the Lower Sand Pit, [insert range and elevation] and
> [insert range and concentration] for samples at the
> Clay Pits, and [insert range and elevation] at the
> Field."
> c. 3 rd sentence. Consider deleting the word
> "natural".

7. References.

> a. Please provide a reference section. Cite
> the cone & quarter technique as well as the Study
> Design Document I prepared for planning and

> implementation of your work (USEPA 2008). I assume
> you still have this doc, but if not, it's posted on
> EPA's Libby website which is located at:
> www.epa.gov/region8/superfund/libby.gov . Please
> list
> other references as recommended above.
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> Hotmail: Trusted email with powerful SPAM
> protection.
> Sign up now.
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> now.
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LETTER

Amphibole forensics: Using the composition of amphiboles to determine their source, the Libby, Montana, example†

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ABSTRACT

As part of a larger on-going research project, we found amphiboles in soils in Libby, Montana, that were derived from both the former vermiculite mine near Libby and other sources. SEM-EDS spectra of the soil amphiboles from these locations are used to ascertain if the amphiboles came from the vermiculite deposit near Libby, Montana, or some other source. This distinction is possible because amphiboles known to originate from the vermiculite deposit all contain Na and K that can be observed in the EDS spectra. We make this statement with confidence because multiple workers have performed EPMA characterization on over 40 amphiboles collected from the deposit and all are found to contain measureable amounts of Na and K. In this brief communication, we show examples from four locations that contain amphiboles consistent in composition and others that have inconsistent composition when compared to reference amphiboles collected from the vermiculite deposit. Also, we demonstrate the presence of amphiboles consistent in composition with amphiboles known to originate from the deposit in sediment that pre-date mining activity. A full-length publication is in preparation detailing the quantity, distribution, and composition of amphiboles in the Libby Valley, if they are consistent in composition with amphiboles known to originate from the deposit or not, and if they pre- or post-date mining operations.

Keywords: Amphibole, amphibole asbestos, Libby, Montana, EPA, SEM-EDS

INTRODUCTION

Amphiboles form in many geologic settings and as such can vary widely in composition and habit. In certain geological settings, it is possible to use amphibole compositions to determine their source. Geologists have used these sorts of methods for years to unravel the geological development of an area. However these methods could also find use where mining and milling processes may have led to alleged soil contamination, as is the case in Libby, Montana. Currently much of the area has been deemed a Superfund site based on amphibole asbestos contamination from the former vermiculite mine (see Gunter et al. 2007 and references therein). The EPA now contends some areas of Libby were contaminated by mine tailings (used on running tracks at two schools) (U.S. EPA 2001) or vermiculite ore used as attic insulation or amendments in gardens and lawns (U.S. EPA 2002). However, Gunter (2008) recently questioned what is and is not contamination in soils in Libby, based on the following: (1) some of the amphiboles might have been naturally transported from the vermiculite deposit and deposited in Libby soil pre-dating mining, and (2) other amphiboles could occur in the soil and sediment whose source is not the vermiculite deposit.

Issues surrounding the former vermiculite mine near Libby, Montana, have been in the national spotlight for almost a de-

cade, mainly due to the amphibole asbestos occurring in the vermiculite ore. Historically, the amphiboles in the deposit were often referred to as tremolite (see Gunter et al. 2007 and references therein). The amphiboles occur in an alkaline-rich pluton (Larsen and Pardee 1929) and thus their increased Na and K content render them as predominately winchite and richterite, with only minor tremolite, and trace magnesio-riebeckite and magnesio-arfvedsonite (Meeker et al. 2003). As part of a much larger project dealing with the legal issues surrounding the deposit and the possible contamination of soils in the town of Libby, we started collecting soil samples in and around Libby to determine the distribution of amphiboles originating from the vermiculite deposit; our goal was to try and distinguish the pre- and post-mining concentration of these amphiboles in the soil. The deposit is upstream from Libby and the rocks composing it are highly erodible (Larsen and Pardee 1929; Boettcher 1967). The area was also glaciated during Pleistocene times (Larsen and Pardee 1929; Boettcher 1967; Smith 2006). Figure 1 shows that the mine is up-river with respect to Libby, and located in a naturally eroded bowl. Our hypothesis was that sediments derived from the deposit would have been transported downstream and deposited in a glacial lake that covered the current location of Libby (Smith 2006); its approximate shoreline is outlined in Figure 1. During our study the EPA also has posed the question about “background” levels of amphiboles from the deposit (U.S. EPA 2008), which they refer to as “Libby amphiboles.” One reason they became concerned was the possibility that these amphiboles could occur in borrow pits where they had obtained “clean” soil to replace the contaminated soil in lawns and gardens in Libby.

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† Open Access: Thanks to the authors’ generous funding, this article is freely available online at MSA and GSW (<http://ammin.geoscience-world.org>). The MSA web site has info about the MSA Open Access policy at http://www.minsocam.org/MSA/ammin/e-pub_policy.htm (in the section called open access and self archiving).

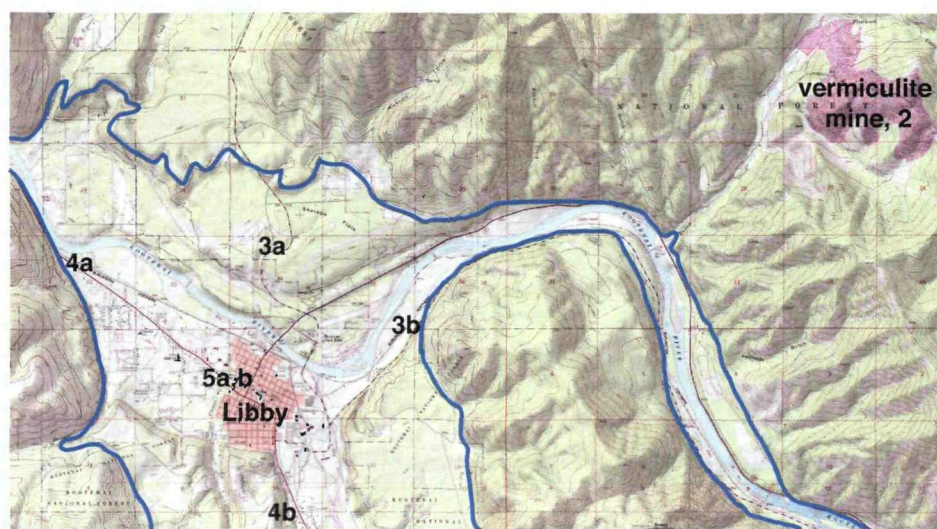


FIGURE 1. A section of a 7.5 min topographic map of Libby, Montana. The blue lines are the approximate boundary of glacial Lake Kootenai. The numbers are sample collection locations keyed to Figures 2–5.

SAMPLE SELECTION AND EXPERIMENTAL METHODS

Sample selection

A series of amphibole samples from the deposit are used as reference materials herein to compare to amphiboles obtained from six soil samples collected at five locations in the Libby area. The soil sample locations are shown on Figure 1 and are referenced to Figures 2–5. Figures 2–5 show amphibole SEM images and EDS spectra for each respective location. The soil samples presented here are a subset of a larger collection of 66 samples from 33 locations, and were chosen to represent the compositions found in the larger suite. The three amphiboles in Figure 2 are from the deposit and represent three species of amphibole: tremolite, winchite, and richterite; note all of which contain observable Na and K in increasing amounts (Sanchez et al. 2008). They were chosen because they represent low, intermediate, and high Na and K content, and are used as our reference amphiboles.

Powder X-ray diffraction

Back-filled cavity mounts were prepared by first sieving the soil to 250 mesh (or $<63 \mu\text{m}$) and then placing 2 g in a McCrone micronizing mill with 25 mL of methanol for 12 min. Data were collected from $2-42^\circ 2\theta$ with a step size of 0.02° and a count time of 9 s. Scans were also made from 9.5 to $11.5^\circ 2\theta$ with a step size of 0.02° and a count time of 180 s to record the region of the 110 amphibole peak. We used the Rietveld method on the $2-42^\circ 2\theta$ scans to determine the major mineral phases and developed a set of standards (i.e., samples with known added amount of amphibole) for the 9.5 to $11.5^\circ 2\theta$ to obtain detection levels of 0.1% or better (Gunter et al. 2008); however, this method yields only amphibole concentrations and cannot distinguish the compositions of the amphiboles.

Scanning electron microscopy and energy dispersive spectroscopy

Soil samples were prepared for SEM by sieving to 250 mesh (or $<63 \mu\text{m}$), placing a small amount of material on an aluminum SEM stub covered with carbon tape, and applying a carbon coating. For each elongated mineral particle observed in the SEM, an EDS spectrum was collected at 20 kV accelerating voltage.

RESULTS AND DISCUSSION

We are only reporting herein a small subset of our results from this project, while the larger data set is being analyzed. We feel these results are time-sensitive for Libby, as well as other locations where amphibole asbestos “contamination” may be incorrectly attributed to human activity when natural processes are to “blame,” or the offending amphiboles may have come

from a non-mining source. As stated in the introduction, our major objective in this study was to determine the amphibole content in soils in the Libby area and to determine what portion of the amphibole were pre- or post-mining (i.e., compare the natural occurrences vs. the contamination from mining). We found all 66 of our samples to contain amphiboles, based on powder X-ray diffraction from tenth’s of a percent to several percent (Gunter et al. 2008). What we should have, but did not anticipate was that the majority of the amphiboles (~90%) were not from the Libby vermiculite deposit. The amphiboles originating from the

deposit can be distinguished from those not originating from the deposit based on the fact that all of the analyzed amphiboles from the deposit contain Na and K, and Na and K peaks can be seen in the SEM-EDS spectra on amphiboles obtained from soil samples. The ability to use EDS to aid in identification of these amphiboles was also pointed out in Bern et al. (2002) and Eckberg et al. (2007).

Figure 1 shows the distribution of the samples in this study as well as the location of the mine and Libby. Figure 2 shows representative samples from the mine. (Details of the samples are given in the figure captions, and EPMA data are given in Sanchez et al. 2008.) They were selected to show low, intermediate, and high Na and K levels. Wylie and Verkouteren (2000), Meeker et al. (2003), Gunter et al. (2003), and Bandli et al. (2003) all present EPMA data that are in the range of the EDS spectra shown here, with the exception of sample 20 in Meeker et al. (2003) that has a $\text{Na} + \text{K} = 0.5$, being slightly lower than our sample shown in Figure 2a [where $\text{Na} + \text{K} = 0.7$, Sanchez et al. (2008), Table 2b therein]. Of the 43 samples for which EPMA data are available, 0.5 is the lowest and 0.7 is second lowest value. Note all of these spectra were collected on polished samples prepared for EPMA analysis. For the amphibole particles obtained from the soil samples, this is not possible as the samples are too small.

Figure 3 shows two high-aspect ratio amphibole particles; both were collected from lake sediment in road cuts as noted on Figure 1. In each case, we dug back into sediment so as to obtain an undisturbed, natural sample. The particle in Figure 3a has a composition inconsistent with amphibole originating from the deposit, while the particle in Figure 3b is consistent with amphibole originating from the deposit. Figure 4 shows two more amphibole particles that are slightly wider than the ones shown in Figure 3. The deposit amphibole in Figure 4 was collected in lake sediments in a road-cut, while the non-deposit amphibole was collected with a hand auger at a depth of ~1 m, both would represent a pre-mining time period. Notice how the samples in Figures 3 and 4 were collected around Libby; the

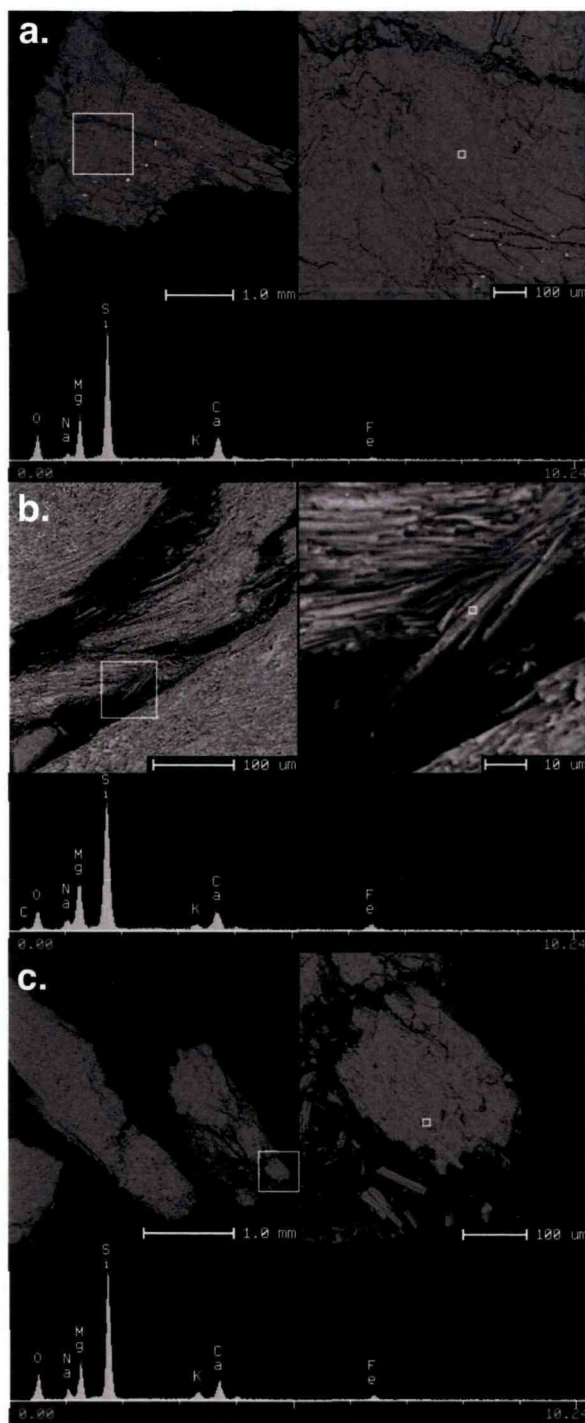


FIGURE 2. Three sets of SEM images and associated EDS spectra obtained on polished samples of amphiboles from the former vermiculite mine near Libby, Montana: (a) Butte 2, (b) float, and (c) Harvard 1. (Names correspond to those used in Sanchez et al. 2008.) For each, the left image is at lower magnification and contains a box that represents the higher magnification image to the right. The small box on the right image coincides to the locations of the SEM beam, and in turn, the area analyzed in the EDS spectra immediately below the images. Note how the Na and K contents increase from a to b to c.

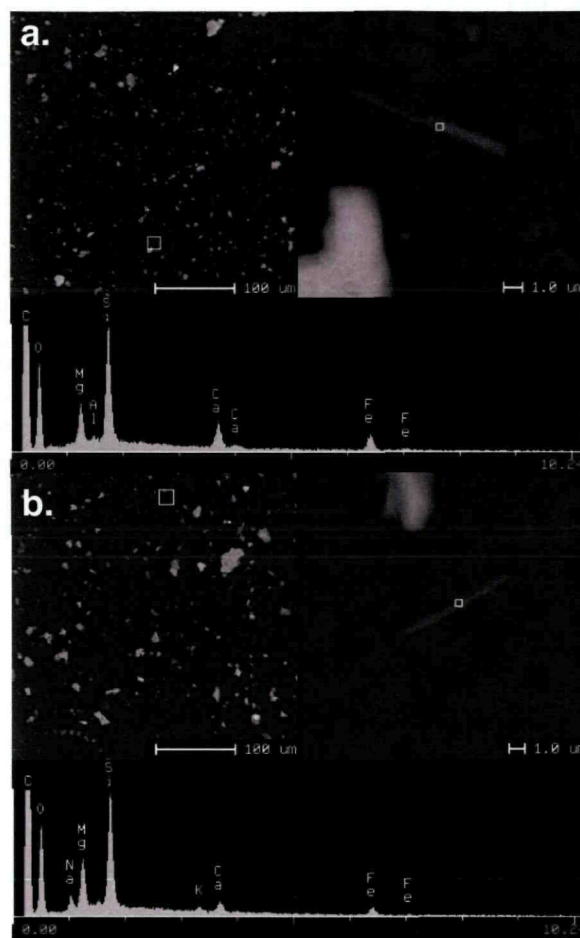


FIGURE 3. Two sets of SEM images and associated EDS spectra obtained on amphibole particles collected in road cuts of lake sediments located at 3a and 3b on the map in Figure 1. Note the upper EDS spectra is Na and K free, while the lower one contains Na and K, and resembles the EDS spectra shown in Figure 2.

sample in Figure 5 was collected in town just below the grass roots in a schoolyard. Note here that, again, the upper sample is Na and K free (i.e., it has a composition inconsistent with those from the deposit), while the lower sample contains Na and K and has similar composition to those from the deposit. Thus, our results to date demonstrate the ability to distinguish particles with compositions consistent with reference amphiboles obtained from the former vermiculite deposit from amphibole particles with compositions inconsistent with those known to be from the deposit.

ACKNOWLEDGMENTS

We thank Bryan Bandli and Antonio Gianfagna for helpful reviews. We also thank W.R. Grace Co. for partially funding this work.

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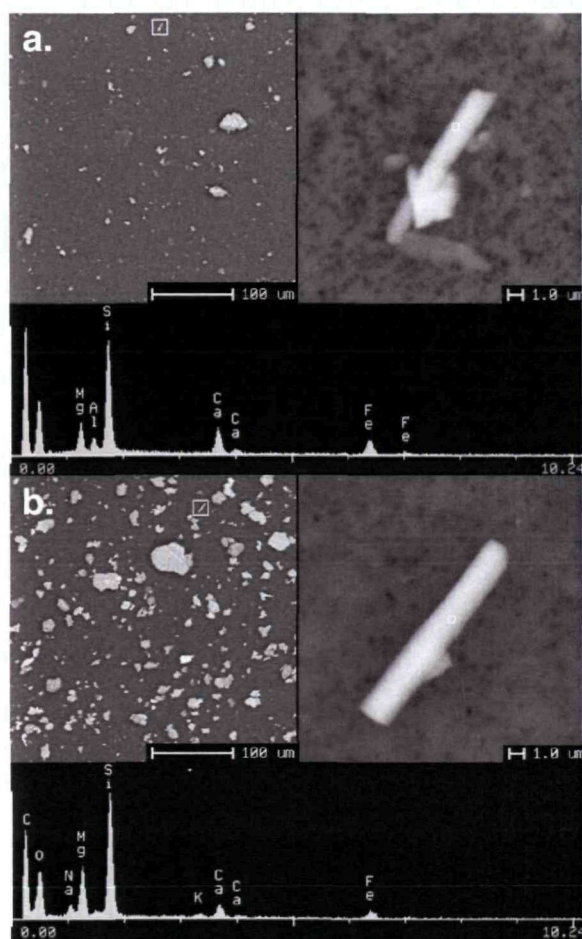


FIGURE 4. Two sets of SEM images and associated EDS spectra obtained on amphibole particles occurring at 4a and 4b on the map in Figure 1. These samples have similar EDS spectra to those shown in Figure 3, but the particles are wider. The upper EDS spectra is Na and K free, while the lower one contains Na and K and resembles the EDS spectra shown in Figure 2.

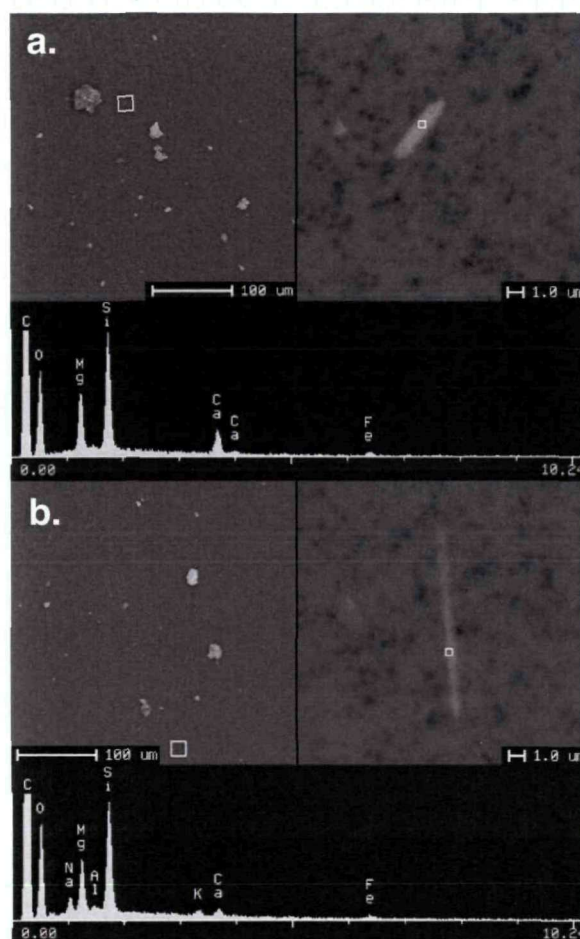


FIGURE 5. Two sets of SEM images and associated EDS spectra obtained on amphibole particles collected at location 5a and 5b on the map in Figure 1. The upper amphibole is not associated with the vermiculite deposit, while the bottom one is, based on the Na and K contents.

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